INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON



# **Experiment Report Form**

# The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:** 

http://193.49.43.2:8080/smis/servlet/UserUtils?start

#### Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

#### Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

#### **Published** papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

## **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

### **Instructions for preparing your Report**

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	<b>Experiment title:</b> Density of carbonatite melts by X-ray radiography: implications for mantle metasomatism and the global cycle of carbon	<b>Experiment</b> <b>number</b> : HS-4349
Beamline:	Date of experiment:	Date of report:
ID27	from: 09/06/2011 to: 14/06/2011	26 <sup>th</sup> April . 2012
<b>Shifts:</b> 15	Local contact(s): Sylvain Petitgirard	Received at ESRF:
Names and affiliations of applicants (* indicates experimentalists):		
Carmen Sanchez-Valle <sup>1</sup> *, Sujoy Ghosh <sup>1</sup> and Sylvain Petitgirard <sup>2</sup>		
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#### **Report:**

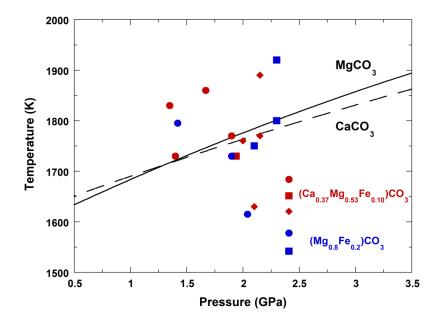
Although carbonate melts are volumetrically minor phases in the mantle, they may control the mobility of C and its residence time in the mantle, ultimately contributing to the gobal carbon cycle. Carbonate melts are also considered as effective metasomatic agents because of their wetting properties, high migration rate and characteristic trace element enrichment. The density of carbonate liquids is thus an important parameter to model their percolation through the mantle and evaluate their behavior as metasomatic agents and carbon reservoirs, but available data remains scarce at relevant P-T conditions and melt compositions.

During the allocated beamtime at ESRF ID27, experiments were performed using a panoramic Paris-Edinburgh Press (PE Press) to determine the density of carbonate melts in the ternary system CaCO<sub>3</sub>-MgCO<sub>3</sub>-FeCO<sub>3</sub> as a function of pressure (1.42 to 2.30 GPa) and temperature (1630 to 1920 K) (Figure 1). Specifically, we performed a total of 5 suscessfull experiments to determine the density of (Mg<sub>0.8</sub>Fe<sub>0.2</sub>)CO<sub>3</sub> and (Ca<sub>0.37</sub>Mg<sub>0.53</sub>Fe<sub>0.10</sub>)CO<sub>3</sub> liquids at upper mantle conditions. The later composition is representative for melts produced from the partial melting of carbonated peridotites in the upper mantle.

High pressure and temperature experiments were performed in a panoramic PE press, using 7 mm tungsten carbide anvils. The sample containers consisted of natural single crystal diamond cylinders (Almax Industries, Belgium) with  $\emptyset$ in = 0.5 mm,  $\emptyset$ out = 1.5 mm and a height of 1.0 mm. The capsule was sealed on both sides by a thin Rhenium (50 µm) to prevent Fe loss through alloying and Pt disks (200 µm) where added on top to ensure compresson of the charge. The assembly was enclosed in an hBN cylinder and two hBN caps were placed on both ends acting as pressure-transmitting medium and surrounded by a graphite heater. The assembly was placed inside a standard 7 mm boron gasket and a plastic supporting ring was added around the girdle of the gasket to increase the stability of the assembly. This modification of the gasket assembly allowed to reduce the number of failed experiments to30% compared to the 50% rate usually obtained (see experimental report HS-4216).

The absorption scans were collected with two ionization chambers and at a relatively low X-ray energy (Mo edge, 20.0 keV) for an optimal absorption contrast. Two different pressure marker (hBN and Pt) were used to determine the pressure and temperature by X-ray diffraction from the respective equations of state. After verification of the liquid state of the sample by X-ray diffraction, absorption scans of the

assembly were collected at various P-T conditions (Figure 1). Typically, 20 scans were collected at each P-T in different positions of the sample to increase the precision in the determination of the density. All absorption scans were of excellent quality and allow the accurate determination of the absorption coefficient and density at each investigated P-T conditions.



**Figure 1.** Summary of density measurments in carbonate  $[(Ca_{0.37}Mg_{0.53}Fe_{0.10})CO_3 \text{ and } (Mg_{0.8}Fe_{0.1})CO_3]$  liquids performed during experiment HS-4349. Different symbols correspond to different runs. Solid and dashed lines represent respectively the melting curve of MgCO<sub>3</sub> and CaCO<sub>3</sub>.

Data processing is in progress to determine the density and to parameterize the equation of state for carbonate melts at crustal and upper mantle conditions. Run products have been recovered and are being characterized for the Fe content using electron microprobe at ETH Zurich and for the  $CO_2$  content by Secondary Ion Mass Spectrometry (SIMS) at ISEI, Okayama University (coll. Eizo Nakamura). Preliminary results show only minor changes in the chemistry of the samples during the run.

These experiments provide the first experimentally measured density dataset for melts of representative compositions for natural carbonatites at pressure-temperature conditions of geological relevance. These results will further allow establishing buoyancy relations with mantle rocks and their evolutions with pressure to better quantify the extraction of C-bearing liquids from the residual rocks during partial melting and the ascent of melts through the mantle and their role as metasomatic agents in the upper mantle. Preliminary results of this study will be presented at the Goldschmidt conference 2012 in Montreal in June 24-29<sup>th</sup> and a manuscript reporting the density of carbonatite melts will be submitted next Fall.